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DIAMAGNETIC ANOMALY IN PRESSURE QUENCHED CDS.(U)
MAY 79 C G HOMAN, D P KENDALL

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DIAMAGNETIC ANOMALY IN PRESSURE QUENCHED CdS

C. G. Homan
D. P. Kendall

May 1979

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Compacted powder samples of CdS, pressure quenched at approximately 10^6 bars/sec at room temperature from pressure above the semiconducting to conducting transition pressure (about 40 kilobars) exhibit an ac diamagentic anomaly and an increase in electrical conductivity in the diamagnetic state at temperatures near 150K. These samples, as well as samples pressure quenched from lower pressures but above 10 kbars reveal a layered structure consisting of amorphous platelets in a compact powder matrix. Other changes in the physical properties of these samples are reported.

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INTRODUCTION

Recently, Russian workers have reported a diamagnetic state with susceptibility approaching $\chi = -1$ accompanied by increased electrical conduction in the diamagnetic state in CuCl samples near 107K when specially prepared samples are temperature quenched at constant pressures above 5 kbars.[1] Chu et al[2] have independently reported an anomalous ac magnetic transition from a normal to diamagnetic to normal state accompanied by increased electrical conduction in the diamagnetic region occurring at temperatures above 90K when CuCl samples are rapidly warmed at pressures below 32 kbars. Tentative confirmation of the Russian results have been reported by Lefkowitz et al.[3]

Other results include our observation of massive precipitation at pressures of approximately 50 kbars, which we related to the disproportionation reaction.[4] Independently, Blount and Phillips predicted that disproportionation would lead to free Cu precipitating in Gunnier-Preston zones.[5] Also, Piermarini and Block have observed that the non-hydrostaticity of the pressure environment plays a critical role in the formation of high pressure phases in CuCl.[6]

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[1] N. B. Brandt, S. V. Kuvschinnikov, A. P. Rusakov, M. V. Semenov, JETP Ltrs, 27, 37 (1978).
[2] C. W. Chu, A. P. Rusakov, S. Huang, S. Early, T. H. Geballe, C. Y. Huang, Phys. Rev. B18, 2116 (1978).
[3] I. Lefkowitz, J. S. Manning, P. E. Bloomfield, Private Communication.
[4] C. G. Homan, to be published VII ARRPT Conference, France, 1979.
[5] E. I. Blount, J. C. Phillips, In press, J. Less Common Metals.
[6] G. Piermarini, S. Block, Private Communication.

The theoretical models proposed by Mott,[7] Halperin and Rice,[8] Herring,[9] Abrikosov,[10] as well as the proximity models of Alexander, Bray and Bardeen[11] suggested to us that a generalized superconductivity behavior may exist for excitonic solids. The present experiments were performed to study this possibility.

APPROACH TO THE PROBLEM

CdS was chosen as a prototype excitonic II-VI compound[12] and to avoid problems of disproportionation. Early resistance measurements by Samara and Drickamer[13] indicated a metallic-like transition near 40 kbars at RT. Subsequent optical measurements[14] and x-ray measurements[15] by Drickamer and coworkers suggest that this transition occurred in the 25-30 kbar region. Our own resistance data suggested that the transition to the high pressure phase occurred near 40 kbars and that the conductivity was several orders of magnitude greater than that obtained in CuCl.[16] We also observed that CdS deviated strongly from the Phillips Van Vechten theory of ionicity.[17]

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[7] N. F. Mott, Phil Mag 6, 287 (1961).
[8] B. I. Halperin, T. M. Rice, Rev. Mod. Phys. 40, 755 (1968).
[9] C. Herring, c.f. ref. 7.
[10] A. A. Abrikosov, In press, JETP Ltrs, 1978.
[11] D. Allender, J. Bray, J. Bardeen, Phys. Rev. B7, 1020 (1973).
[12] C. Kittel, Introduction to Solid State Physics, (John Wiley & Son,NY, (1976), p. 328.
[13] G. A. Samara, H. G. Drickamer, J. Phys. Chem. Solids 23, 457, (1962).
[14] A. L. Edwards, H. G. Drickamer, Phys. Rev. 122, 1149 (1961).
[15] G. K. Lewis, E. A. Perez-Albuerne and H. G. Drickamer, J. Chem. Phys. 45, 598 (1968).
[16] D. P. Kendall, C. G. Homan, Unpublished data on wide variety of excitonic solids. See ref. 19 and 21.
[17] J. A. Van Vechten, Phys. Rev. 182, 891 (1969). Also see the numerous references to J. C. Phillips and coworkers contained in ref. 5.

Pellet polycrystalline samples were prepared by compacting optical grade CdS[18] to about 2 kbars in a piston cylinder device. These samples, which were orange in color were placed in a gasketed Bridgeman anvil pressure device, which is an unsupported version of the pressure device previously reported.[19] The die ring, is not externally supported as before, and a new pyrophylite gasket design allows samples initially, 0.1 inch in diameter by 20 mils thick, to be pressurized. Prior to each experiment the samples are further compacted in situ by slowly loading the anvils to a pressure of about 10 kbars and slowly unloading.

Load is applied to the pressure device by means of a yoke type loading frame mounted in a servo hydraulic tensile testing machine. Cryogenic temperature can be achieved at the sample using a standard liquid He tensile testing cryostat. The specimen resistance is determined by measuring the resistance across the anvils and sample. The anvils are insulated from the loading frame. When CdS samples are pressurized in this device at room temperature, a resistometric trace similar to that shown in Figure 1 is obtained.

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[18] Samples were prepared from ultra-pure optronic grade CdS obtained from Alpha Inorganics, Stock No. 20130, compacted in a small piston cylinder device to a pressure of about 2 kbars. Samples were found to be nearly at theoretical density by a gravimetric technique. Spectrographic analysis by the spark emission method and a quantitative chemical analysis of our sample indicated the following metallic impurities: Fe = 12 ppm, Mg = 3.6 ppm, Cu = 4.2 ppm, Ag = 3.5 ppm, Bi < 1 ppm, Not detected, Ni < 1 ppm, Mn < .46 ppm, Pb < .07 ppm, Al < 1.4 ppm, Cr < 2.8 ppm.

[19] D. P. Kendall, P. V. Dembowski, T. E. Davidson, Rev. Sci. Instrum. 46, 629

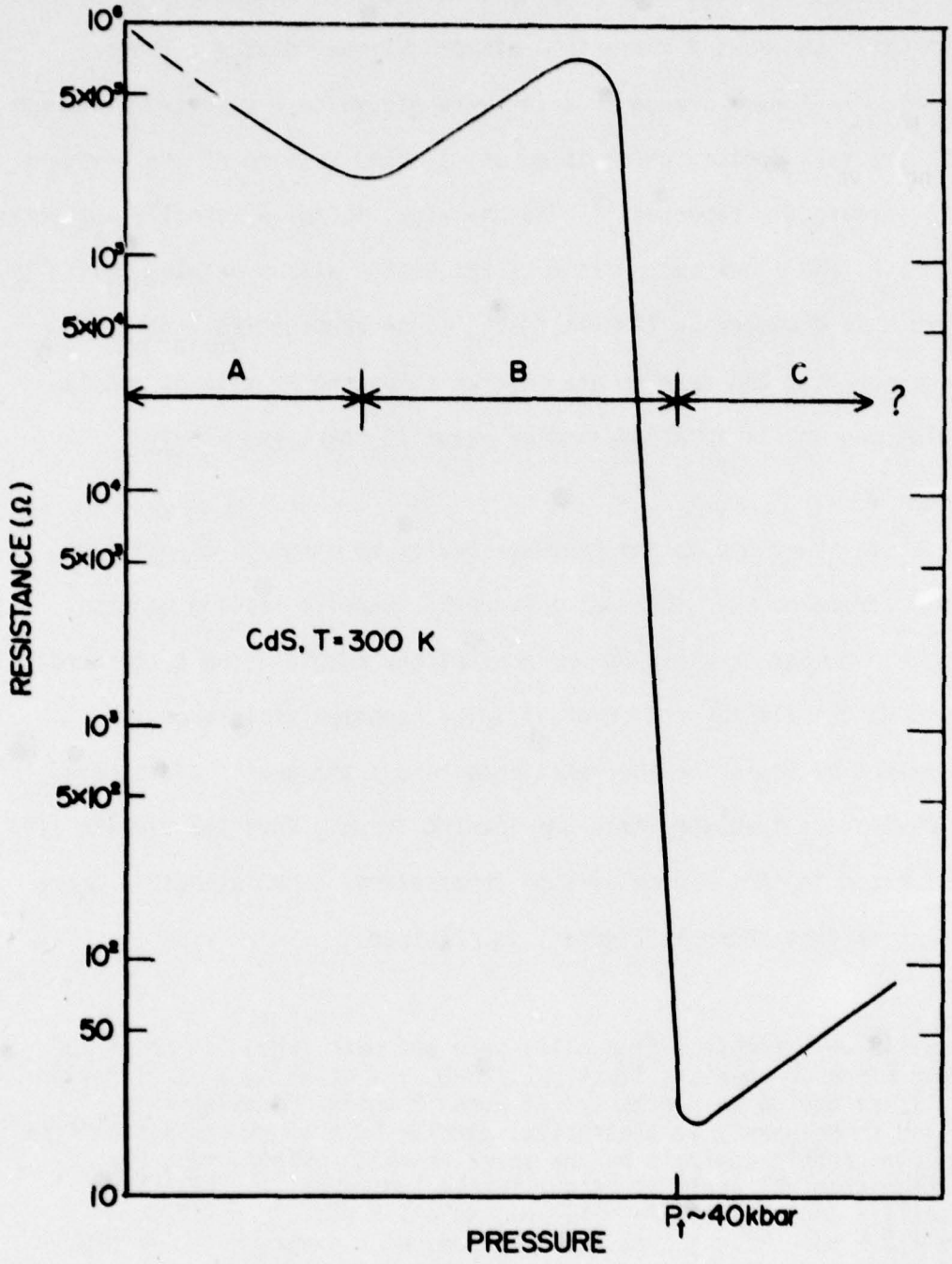

Fig. 1. Typical resistometric trace of CdS at ambient temperatures. See text for description of Regions A, B, and C.

After the various pressure and temperature cycles performed, the samples were removed from the pressure cell at RT and placed in one leg of a mutual inductance bridge. In some of these runs, a sample of Pb having a volume equal to about 10 percent of the CdS samples was placed in the same leg of the bridge. The bridge was excited at 17 Hz and the output of the bridge was determined with a lock-in amplifier. The bridge was balanced by zeroing both the inductive and resistive components of the output.

Prior to the actual magnetic experiments the coil assembly without CdS samples but with the Pb specimen, was cooled to LHe temperature and the bridge balanced. The bridge output was continuously monitored as the temperature increased. The Pb transition was clearly seen near 7K, and after rebalancing near 20K, a continuous gradual change in the output occurred which was attributed to thermal effects of the coil and Pb calibrator. However, there are no abrupt discontinuities in either the resistive or inductive components of the output between 4.5K and 240K. The total change in the output over the range of 20K to 240K was 20 percent of the unbalance which would be caused by a superconducting transition of a Pb sample of the same volume as the CdS samples used.

The various CdS specimens were slowly pressurized into each of the three regions shown in Figure 1 (A, B, and C) and pressure quenched to room pressure at RT. Pressure quench rates varying from 10^3 bars/sec to 10^6 bars/sec were used.

Optical microscopy, including polarized light examination and microhardness, scanning electron microscopy, and through transmission Laue x-ray measurements were made at room pressure for various samples. Experimental details will be described separately.[20]

RESULTS

REGION A - There was no discernable change in the measured properties after pressure treatments except for a slight irreversible change in color towards the red. The samples remained optically opaque in the visible and the resistance recovered. No magnetic anomaly was observed from RT to LHe temperatures.

REGION B - In the range of pressure quench rates investigated, there was a definite change in color to a translucent red with domains of opaqueness in the interior of the samples as observed by white light illumination. These opaque domains were not of sufficient extent as to block light transmission through the sample. The volume fraction of opaque domains approached 10 percent when quenched from a pressure near but below the semiconductor-conductor transition pressure (Pt) at room temperature.

Optical and SEM examination revealed a layered morphology, the opaque domains existed in platelets of varying thickness imbedded in a powder compact matrix. These platelets lay in the plane of the anvil faces and generally did not show optical rotation in Nicol polarizers although some small regions of the platelets did rotate as did the matrix material.

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[20] C. G. Homan, D. P. Kendall, J. Barrett, G. Capsimalis, P. J. Cote, L. V. Meisel, to be submitted.

Careful examination at magnifications to 17,000X revealed no evidence of grains in the platelets and the appearance of a broad diffracted halo in the x-ray measurements suggest the amorphous nature of the platelets. However, microcrystallinity cannot be excluded until the radial distribution function determination is made. Microhardness measurements indicate that the glassy platelets were harder and more brittle than the matrix material and were fractured in the transverse plane.

These samples showed no evidence of a magnetic anomaly in the temperature range from 4.5 to 240°K.

Samples pressurized into region B and cooled to LN_2 temperatures at constant load and those samples pressure quenched at RT and subsequently cooled exhibited a negative coefficient of resistance suggesting the semiconducting natuwe of the new domains.

REGION C - Of the several samples pressure quenched from above Pt at room temperature, at rates approaching 10^6 bars/sec, all had a metallic luster and were opaque to transmission of white light. Optical microscopy indicated that those samples quenched from about 50 kbars had an increased volume fraction of platelets approaching 15 percent, nearly all of which were brightly reflecting, imbedded in a dark red powder compact matrix.

Samples held at a constant load above Pt and RT and then cooled to LN_2 temperature exhibited a positive coefficient of resistance and a transition to a more highly conducting state at temperatures as high as 150K. Samples pressure quenched from above Pt at RT to a pressure of about 8 kbar exhibited a resistance increase to 4000 Ω,

(at least three orders of magnitude more conducting than unquenched samples). Subsequent cooling at constant load to LN_2 temperatures showed a sharp increase in conduction occurring near 150K. On warming from LN_2 temperature, the reverse transition occurred near 120K. Recooling the sample from about 150K, produced a similar increased conduction near 100K which recovered on warming near 110K. The apparent reversal in the transition and recovery temperature is attributed to the large difference in cooling rates between those two runs. More significance can be attached to the relative location of the transition recovery temperatures, as the warming rates were essentially the same (about 2 K/min). Finally, samples quenched to room pressure, whose resistance was in the order of 10^4 ohms were cooled to LN_2 and a small increase in conduction occurred at temperatures near 150. The relationship between overall sample resistance and the resistance of the glassy phase is complicated by the presence of the compact matrix material. Therefore, the relative contribution of the glassy platelets and the matrix phase to the above conductivity results cannot be accurately determined. However, a simple network analysis suggests that the glassy metallic phase increased its conductivity by more than an order of magnitude in the transition region.

Magnetic measurements in samples fast pressure-quenched to RP indicated a diamagnetic anomaly near 150K following cooldown to LN_2. Figure 2 is a plot of relative permeability on a sample cooled to LHe temperature as a function of temperature using the Pb sample technique previously described. The magnitude of the unbalance of the bridge

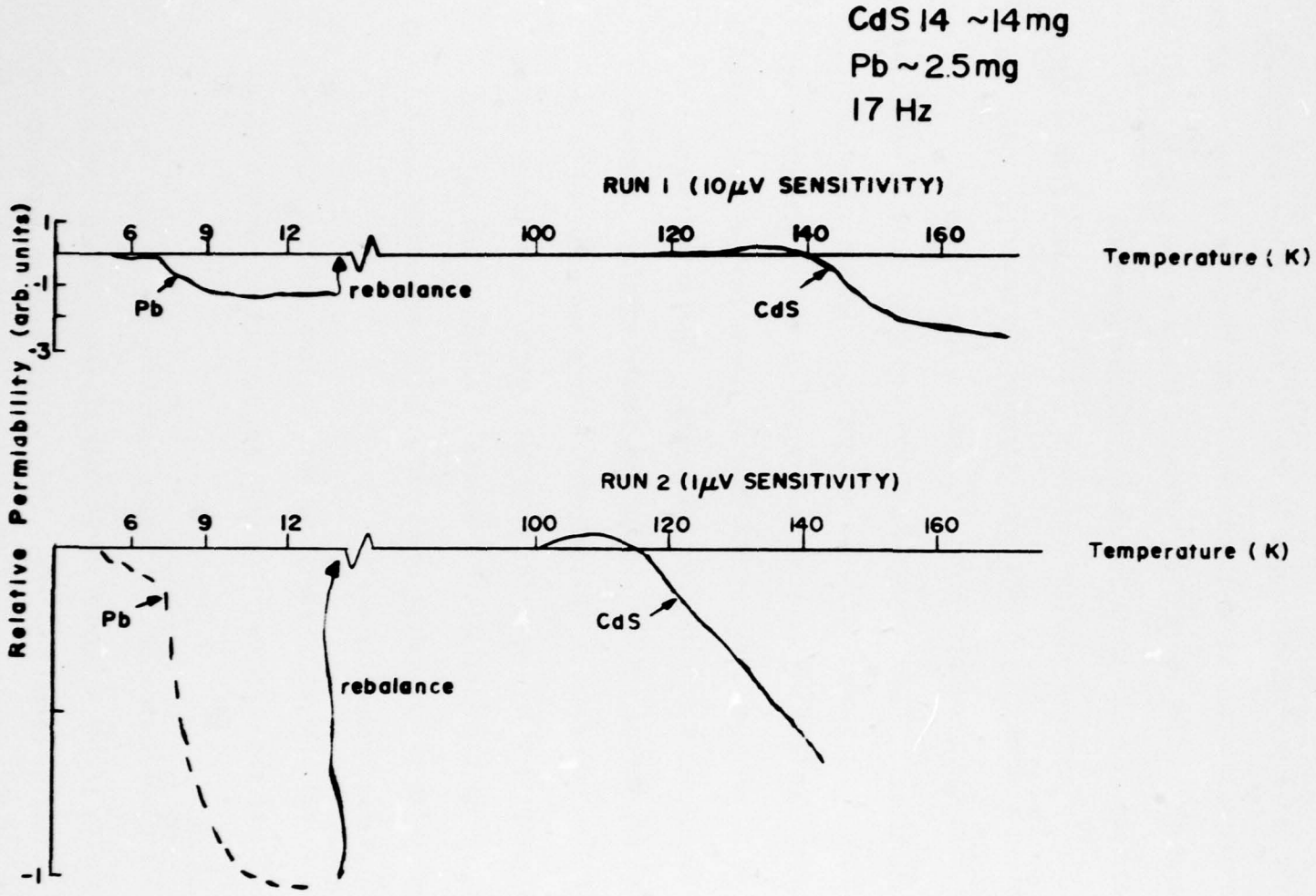

Fig. 2. Relative permeability of a CdS sample quenched from above Pt with a quench rate-10^6 bars/sec. Measured at RP while sample is warming from LHe temperatures. The signal from a Pb calibration pellet is also shown (see text). Temperature uncertainty is $\pm$ 2 K.

suggests that the diamagnetic signal was equivalent to 10 to 15 percent of that for a perfect diamagnet in the CdS sample volume examined, which may relate to the 10-15 percent volume fraction of the new phase. Temperature cycling up to 240K indicated the same decrease in transition temperature as observed in the resistance measurements.

Slowly pressure quenched specimens ($\approx 10^3$ bars/sec) from above Pt, exhibited a morphology, resistance and magnetic behavior similar to those observed in samples pressure quenched from region B.

STABILITY - Those samples fast quenched from above Pt exhibited the diamagnetic anomaly after being exposed to ambient room conditions for up to one hour while being transferred to the inductance bridge. Samples of pressure quenched CdS from all three regions were stored in a RT vacuum dessicator for several months. Re-examination on the optical microscope and SEM yielded the same morphology observed originally. However, an obvious color change had occurred in the powder compact matrix material from red towards the original orange color. X-ray examination also revealed a fading of the halo and strengthening of the powder crystalline lines suggesting some recovery in the glassy regions as well.

The implications of the above observations and the longer term stability of the diamagnetic anomaly are subject to current study.

CONCLUSIONS

The novel technique of pressure quenching polycrystalline compact CdS samples yields a layered morphology of glassy domains imbedded in a polycrystalline compact matrix. These glassy domains appear to be

metallic after quenches from above Pt for quench rates up to 10^6 bars/sec. The optical behavior of our pressure quenched samples closely follow the optical behavior found at pressure for CdS by Edwards and Drickamer.[14]

The material quenched from above Pt transforms at temperatures near 150 K to a more conducting diamagnetic state. A correlation between the volume fraction of the glassy material and the magnitude of the diamagnetic signal is evident.

It is not apparent what role the layered morphology or the amorphous nature of the layers plays in this anomalous behavior, although it is probable that amorphous semiconductors have a smaller band gap than crystalline semiconductors. Causal relationships between these striking structural features and this behavior will be investigated by physically separating the domains from the compact matrix.

The role of temperature quenching in producing those anomalies in our samples is not clear, although in the temperature quench rate range studied, no discernable effect was observed.

It is tempting to label this type of behavior as evidence for high temperature superconductivity. We prefer to await the results of critical field measurements in progress, and to examine the possible explanations by previous theories[7-11] in the light of this new experimental approach.

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[7]N. F. Mott, Phil Mag 6, 287 (1961).
[8]B. I. Halperin, T. M. Rice, Rev. Mod. Phys. 40, 755 (1968).
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[10]A. A. Abrikosov, In press, JETP Ltrs, 1978
[11]D. Allender, J. Bray, J. Bardeen, Phys. Rev. B7, 1020 (1973).
[14]A. L. Edwards, H. G. Drickamer, Phys. Rev. 122, 1149 (1961).

Finally, close examination of our own body of experimental data[16,19,21] and the extensive literature data suggests that similar anomalies may exist in many other I-VII; II-VI and IV-V compounds.

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[16] D. P. Kendall, C. G. Homan, Unpublished data on wide variety of excitonic solids. See

[19] D. P. Kendall, P. V. Dembowski, T. E. Davidson, Rev. Sci. Instrum. 46, 629

[21] C. G. Homan, D. P. Kendall, T. E. Davidson, J. Frankel, Sol. St. Comm. 17, 831 (1975).

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